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Indian Standard

METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 4 DETERMINATION OF NICKEL-DIMETHYLGLYOXIME PHOTOMETIRC METHOD

(First Revision)

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PART 4 DETERMINATION OF NICKEL-DIMETHYLGLYOXIME PHOTOMETIRC METHOD

(First Revision)

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METHODS OF CHEMICAL ANALYSIS OF BRONZES

PART 4 DETERMINATION OF NICKEL-DIMETHYLGLYOXIME PHOTOMETRIC METHOD

(First Revision)

0. FOREWORD

0.1 This Indian Standard (Part 4) (First Revision) was adopted by the Bureau of Indian Standards on 22 July 1987, after the draft finalized by the Methods of Chemical Analysis of Non-ferrous Metals Sectional Committee had been approved by the Structural and Metals Division Council.

0.2 IS : 4027 first published in 1967, covered determination of copper, lead, tin, manganese, phosphorus, nickel, iron, silicon, aluminium, zinc and antimony in bronzes. While reviewing this standard, the Sectional Committee decided that it is convenient to revise this standard in series of parts which, on publication will supersede the relevant method for determination given in IS : 4027-1967*. This part is one of that series and covers the determination of nickel by dimethylglyoxime photometric method. The other parts are as follows:

Part 1 Determination of copper and lead by electrolytic method

Part 2 Determination of manganese by photometric method

Part 3 Determination of phosphorus by volumetric method

Part 5 Determination of tin by iodimetric method

Part 6 Determination of zinc by complexometric (EDTA) method

Methods for chemical analysis of other constituents in bronzes, namely, aluminium, iron, silicon and antimony are under preparation, and will be published in subsequent parts of above series.

0.3 The method for determination of nickel by the dimethylglyoxime (gravimetric) method, stipulated in earlier edition, has been substituted by photometric method.

0.4 The method of analysis prescribed in this standard may primarily serve as referee method and may also be used by the laboratories for their day-to-day work. Due consideration has been given in the preparation of this standard to the facilities available in the country for such analysis.

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*Methods of chemical analysis of bronzes.

0.5 In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, shall be rounded off in accordance with IS : 2-1960*.

1. SCOPE

1.1 This standard (Part 4) prescribes a method for determination of nickel in the range as specified in the relevant Indian Standards on bronzes.

Note — This method is not applicable to aluminium bronze and aluminium-silicon bronzes where nickel is 4 to 5 percent.

2. SAMPLING

2.1 Samples shall be drawn and prepared in accordance with IS : 1817-1961†.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, analytical grade reagents and distilled water (*see* IS : 1070-1977‡) shall be used.

4. DETERMINATION OF NICKEL BY DIMETHYLGLYOXIME PHOTOMETRIC METHOD

4.1 **Outline of the Method** — Nickel in higher oxidation states forms a red coloured soluble nickel salt with dimethylglyoxime. Photometric measurement is made at 530 nm.

4.2 **Concentration Range** — The recommended concentration range is from 0·05 to 1 mg of nickel in 100 ml of solution.

4.3 **Interfering Elements** — Copper and manganese interfere. Cobalt may interfere, if present in amounts approximating the nickel content.

4.4 **Special Apparatus** — Electrodes for electroanalysis.

4.5 Reagents

4.5.1 *Concentrated Nitric Acid* — r.d. = 1·42 (conforming to IS : 264-1976§)

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*Rules for rounding off numerical values (*revised*).

†Method of sampling non-ferrous metals for chemical analysis.

‡Specification for water for general laboratory use (*second revision*).

§Specification for nitric acid (*second revision*).

4.5.2 *Hydrofluoric-Boric Acid Mixture* — Add 200 ml of hydrofluoric acid to 1 800 ml of a saturated solution of boric acid and mix. This mixture may be stored in a glass bottle.

4.5.3 *Citric Acid Solution* (100 *g/l*) — Dissolve 100 g of citric acid in water and dilute to 1 litre. The addition of 1 g of benzoic acid will prevent bacterial growth.

4.5.4 *Bromine Water* — Saturated.

4.5.5 *Dilute Ammonium Hydroxide* — (1:1) (*v/v*).

4.5.6 *Alcoholic Solution of Dimethylglyoxime* — 10 g/l.

4.5.7 *Standard Nickel Solution* (1 *ml* = 0·02 *mg Nickel*) — Dissolve 0·2 g of nickel (purity 99·9 percent, *Min*) in 20 ml of dilute nitric acid (1:1) with the aid of gentle heat. Cool, transfer to a 1-litre volumetric flask, make up to the mark with water, and mix. Dilute 100 ml of this solution to 1 litre in volumetric flask with water and mix.

4.6 Procedure

4.6.1 Transfer 1·000 g of the sample to a 200-ml electrolysis beaker or a 300-ml Erlenmeyer flask.

4.6.2 Dissolve in 15 ml concentrated nitric acid and 15 ml hydrofluoric boric acid mixture. Cover the beaker and add 7 ml of water. Allow to dissolve without heating.

4.6.3 Boil gently to expel brown fumes. Rinse the cover glass and dilute to approximately 175 ml and electrolyse to remove copper.

4.6.4 When deposition of copper is complete, lower the beaker, while rinsing the electrodes with water, if any insoluble material is present, filter and wash with water. Transfer the electrolyte to a 200-ml volumetric flask, make up to the mark and mix.

4.6.5 Transfer a suitable aliquot (that will cover the desired range of nickel) to a 100-ml volumetric flask, and add successively swirling the flask between additions, 5 ml of citric acid, 5 ml of bromine water, and sufficient dilute ammonium hydroxide solution to bleach the bromine. Add 3 ml of dilute ammonium hydroxide solution in excess and cool the flask to room temperature.

4.6.5.1 Add 3 ml of the alcoholic solution of dimethylglyoxime, taking care to complete the addition of bromine water dilute ammonium hydroxide and dimethylglyoxime to a given solution within a period of 5 minutes. Make up to 100 ml, mix and allow to stand for two minutes.

4.6.6 Transfer suitable portion of the solution to an absorption cell and measure the absorbance at 530 nm. Run a blank through the procedure and correct the absorbance of the sample by subtracting that of the blank.

4.6.7 Compute the nickel content from a calibration curve.

4.7 Calibration Curve

4.7.1 Transfer 1·0, 2·0, 4·0, 6·0, 10·0, 15·0, 20·0 and 25·0 ml of nickel solution (1 ml = 0·02 mg Ni) to eight 100-ml volumetric flasks. Add 2 ml of perchloric acid to each flask. Proceed in accordance with **4.6.5**, **4.6.5.1** and **4.6.6**.

4.7.2 Prepare the calibration curve by plotting absorbance against the mass of nickel in the various aliquots after deducting the blank reading.

4.8 Calculation — Calculate the percentage of nickel as follows:

$$\text{Nickel, percent} = \frac{A}{B} \times \frac{1}{10}$$

where

A = mg of nickel found in 100 ml of the final solution, and

B = g of sample represented in 100 ml of the final solution.

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